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Indian Standard

METHOD OF TEST FOR DETECTION AND DETERMINATION OF ANTIOXIDANT ADDITIVES IN INSULATING OILS

UDC 621·315·615·2 : 543 : 66·094·382

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

March 1993 Price Group 2

Fluids for Electrotechnical Applications Sectional Committee, ETD 03

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Fluids for Electrotechnical Applications Sectional Committee had been approved by the Electrotechnical Division Council.

Test for detection and determination of antioxidant additives in insulating oils was part of the specification, IS 335 on new mineral insulating oils.

With the advancement of technology and experience gained, this test method has been developed taking into account various types of inhibitors possible in liquids.

Since this method can also be used to detect inhibitors in used oil also, therefore it was decided to bring out separate standard on this subject.

In the preparation of this standard, considerable assistance has been derived from the following publications:

IEC Pub 666 (1979) Detection and determination of specified antioxidant additives in insulating oils, issued by the International Electrotechnical Commission (IEC).

ASTM D 2668 : 1977 Standard test method for 2, 6 ditertiary butyl para cresol and 2, 6 ditertiary butyl phenol in electrical insulating oil by infra-red absorption, issued by the American Society for Testing and Materials.

AMENDMENT NO. 1 MARCH 1997
TO
IS 13631 : 1993 METHOD OF TEST FOR DETECTION AND DETERMINATION OF ANTIOXIDANT ADDITIVES IN INSULATING OILS

(*Page* 1, *clause* 3) — **Insert the following in the third para:**

'Add 1 ml of alcoholic phosphomolybdic acid solution (5%) and shake.'

(ET 03)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHOD OF TEST FOR DETECTION AND DETERMINATION OF ANTIOXIDANT ADDITIVES IN INSULATING OILS

1 SCOPE

1.1 The methods described are to be used for the detection and determination of antioxidant additives in new hydrocarbon insulating oils. The detection methods are to be applied to assess whether or not a hydrocarbon insulating oil contains an antioxidant additive as specified by the supplier.

The determination methods are used for the quantitative determination of antioxidant additives previously detected by the appropriate detection method.

NOTE — The methods described may also be used for oils in service.

2 REFERENCES

The following Indian Standard is a necessary adjunct to this standard:

IS No.	*Title*
12026 (Part 1) : 1986	Guide for the determination of the aromatic hydrocarbon content of new insulating oils : Part 2 Infrared spectrophotometric method

SECTION I METHODS FOR THE DETECTION OF ANTIOXIDANT ADDITIVES

3 DETECTION OF PHENOL TYPE INHIBITOR

Pour 2 ml ethanol into test tube.

Add 2 ml of test oil and shake the mixture well (30 sec).

Add 1 ml ammonia, shake and allow to settle until phases separate.

If phenol inhibitor is present there will be a blue or blue-purple coloration.

4 DETECTION OF AMINE TYPE INHIBITOR

Pour 2 ml glacial acetic acid into test tube.

Add 2 ml of test oil and shake the mixture well (30 sec).

Add 1 ml of alcoholic phosphomolybdic acid solution, shake and allow to settle.

If amine inhibitor is present, there will be an intense blue or blue-green coloration.

5 BLANK TEST

It is strongly recommended that a blank test be done using the above procedures with a known pure mineral oil The test to detect phenol type inhibitor will produce a slight blue tinge.

The test to detect amine type inhibitor will produce a pale green coloration.

SECTION II METHODS FOR THE DETERMINATION OF SPECIFIED ANTIOXIDANT ADDITIVES

6 DETERMINATION OF 2.6-DI-TERT-BUTYL PARACRESOL BY INFRA-RED SPECTROPHOTOMETRY

6.1 Summary of the Method

This method determines the amount of di-tert-butyl-paracresol (DBPC) in new mineral oils by measurement of the infrared absorption at the (O — H) stretching frequency of hindered phenols. The method gives a quantitative determination of the additional hindered phenols present in the sample oil compared with the base oil. If the original base oil is not available for comparison, the total phenols present can be determined, but the overall precision of the determination may be comparatively reduced. The detection limit will increase accordingly.

This method is applicable for DBPC concentrations in the range 0·02% — 0·90% by mass.

The absorbance of the (O — H) stretching frequency at 3 650 cm^{-1} is measured in 0·5 mm pathlength cells required path length (in other words — "Cell Thickness") be maintained, so as to ensure accuracy and resolution as may be needed, and the DBPC content obtained from a standard calibration graph prepared by adding weighed quantities of DBPC to the original base oil.

6.2 Reagent

—2, di-tert-butyl-paracresol, analytical grade.

NOTE — Reagent used in infra-red spectroscopy must be stored in brown bottles and away from light.

6.3 Equipment

—Double-beam infra-red spectrophotometer having a resolution of better than 5 cm^{-1} at 3 500 cm^{-1} and covering the range 4 000 cm^{-1} to 3 000 cm^{-1}.

—A matched pair of 0·5 mm path length liquid cells with sodium chloride windows.

6.4 Sample Condition

Samples should be shielded from heat and direct sunlight at all times and should be dried by filtering through a dried Whatman No. 1 filter paper before use.

6.5 Calibration

Standard calibration solutions are prepared by dissolving weighed amounts of DBPC in weighed amounts of the dried base oil.

At least five calibration solutions should be prepared covering the renge 0·02% to 0·50% DBPC by mass Intermediate standards can be prepared if necessrry when the approximate concentration of DBPC in the sample is known.

The absorbance at 3 650 cm^{-1} (A_{3650}) of the calibration solutions is measured by the method detailed in Sub-Clause 6·8 and a calibration curve prepared by plotting A_{3650} againt % DBPC content. The calibration should produce a straight line passing through the origin, according to the Beer-Lambert law of absorption:

$$A = \log_{10} I_0/I = KCD$$

where

A = absorbance

I_0 = intensity of incident radiation

I = intensity of transmitted radiation

K = extinction coefficient [constant for (O—H) of DBPC]

C = concentration of DBPC in % by mass

D = cell path-length

since K and D are constant for this determination, A is directly proportional to C.

6.6 Reference Oil

6.6.1 The reference oil should preferably consist of the base oil used to prepare the inhibited oil.

If no base oil is available, a synthetic reference oil can be made from technical white oil and either aromatic extract or a high aromatic oil of electrical quality. The use of such a synthetic reference oil gives a closer approximation to the true DBPC content than if technical white oil alone is used as the reference oil.

6.6.2 *Method of Preparation of Synthetic Reference Oil*

Measure the absorhance at 1 600 cm^{-1} of the sample oil, technical white oil and aromatic extract or high aromatic oil using the method described in IS 12026 (Part 2): 1986. Calculate A_{1600} for each and then add sufficient refined aromatic extract or high aromatic oil to technical white oil to obtain a mixture with A_{1600} (aromatic content) similar to that of the sample oil.

This synthetic base oil can then be used for the reference oil and for making up the series of calibration solutions.

6.7 Procedure

6.7.1 *Matched Liquid Cells*

Prepare two matched liquid cells with path-lengths of 0·5 mm and sodium chloride windows. Fill both cells with the base oil and, with one cell in the sample beam and the other in the reference beam of the spectrometer check that the IR spectrum between 3 800 cm^{-1} and 3 400 cm^{-1} is a straight line. Note the percentage transmittance (95% – 100%).

Exchange the cells, i.e., transfer the cell in the sample beam to the reference beam and the cell in the reference beam to the sample beam. Repeat the spectrum and again obtain a straight line of approximately 95% —100% transmittance.

If the above conditions are not obtained, clean and polish or reject windows that have an absorbance in this region, and repeat the process until a matched pair of cells is obtained. These are then used for all the determinations.

6.7.2 *Measurement*

Fill one cell with base oil and place in the reference beam of spectrophotometer. Fill the other cell with the oil samples in turn and obtain their spectra between 3 800 cm^{-1} and 3 400 cm^{-1} using a low scanning rate.

6.8 Calculation

6.8.1 *Measurement of Absorbance (see Fig. 1)*

Draw a base line as nearly as possible between 3 610 cm^{-1} and 3 610 cm^{-1} and note the percentage transmittance (I_0) at which the base line crosses the 3 610 cm^{-1}.

NOTE — the percentage transmittance at the tip of the peak at 3 650 cm^{-1} (I), then:

$$A_{3650} = \log_{10} I_0/I$$

The perceutage DBPC equivalent to A_{3650} is read from the calibration graph.

6.9 Precision

To be determined but, as a guide, the standard deviation obtained on 3 replicates of four oil samples using base oil as reference, varied between 0·002% and 0·004% by mass DBPC. The tests were made by one operator using one set of apparatus and the limit of detection of the method was found to be 0·02% by mass.

7 DETERMINATION OF AMINE TYPE INHIBITOR

Test method under consideration.

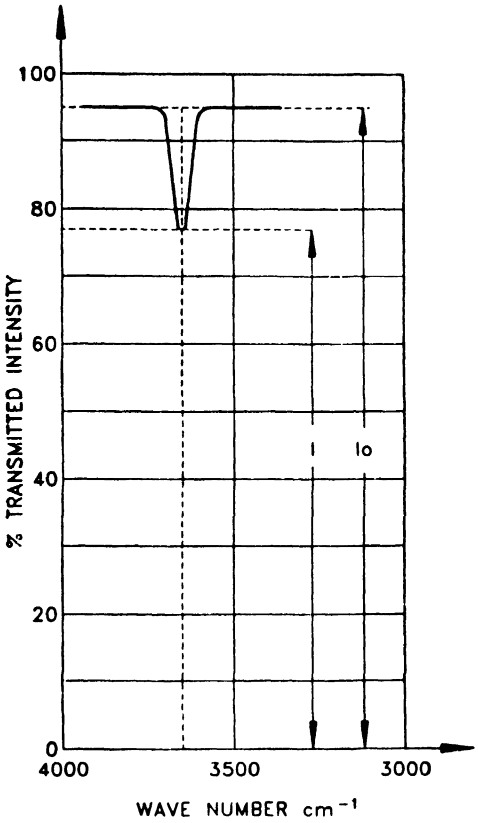

FIG. 1 TYPICAL INFRA-RED SPECTRUM TO DETERMINE DBPC CONTENT

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